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Construction of 300 kV Electron Microscope

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Construction and performance of the 300kV electron microscope which was installed in the Institute for Chemical Research of Kyoto University are described. It has a three stage high voltage generator of cascade type, and each stage is operated at 100kV with a ripple stability less than 1×10^{-4} and is connected to the corresponding electrode of the three stage accelerating gun. The highest tension for the top electrode is applied through an insulated coaxial cable whose exterior surface is kept at 200 kV so that 300 kV is not exposed to the air. This system prevents the accidental corona discharges, and the final stability when operated at 300kV is kept within $3 \times 10^{-4}/\text{min}$.

The best resolution of this electron microscope at 300kV is measured to be about 12\AA .

1. INTRODUCTION

Since the first high voltage electron microscope of 225 kV was constructed by H. O. Müller and E. Ruska¹⁾ in 1941, several high voltage electron microscopes of over 200 kV have been designed and assembled in various countries as follows: von Ardenne²⁾, Zworykin, Hillier and Vance³⁾, van Dorsten and Le Pool⁴⁾, Coupland⁵⁾, Shimadzu, Hori and Kobayashi⁶⁾, Yamaguchi and Shimadzu⁷⁾, and Tadano and Sakaki⁸⁾.

The main purpose of the construction of these high voltage electron microscopes was to obtain the transparent image of thick specimens through which slower electrons can not penetrate. Therefore, in consequence of the developments of specimen preparation techniques such as ultrathin sectioning, shadow casting and replica methods, the efforts directed to increase the speed of electron have not been promoted for more than a decade ever since. However, the necessity to observe specimens of appreciable thickness still remains in major field of electron microscopy; the recent advancement in x-ray microscopy is one of the results of the attempts to fulfill this demand.

In case of organic substances, even specimens thin enough for observation under conventional 50 kV electron microscope always bear the risk of damage due to decomposition caused by inelastic collisions of electrons. Hence, in many cases it is very difficult to assume the intact original molecular arrangements in organic specimen by means of electron diffraction of selected area with highly magnified electron photomicrograph of the same part. Even in case of inorganic substances, the transmission microscopy is always desirable to observe specimens of appreciable mass thickness in order to obtain informations about their three-dimensional structures.

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The best way to minimize the damage of specimen due to electron bombardments and to obtain satisfactory electron diffraction pattern with good penetration is to decrease the cross section of inelastic collision of electrons passing through the specimen, that is, to decrease the wavelength of electrons.

Unfortunately all of the early high voltage electron microscopes described above were abandoned because they were constructed only for investigating the effects of high velocity electrons upon specimens and special concerns were not paid for the practical utilizations. In order to develop a high voltage electron microscope for practical use, the construction of a new 300kV instrument was planned by the present authors in 1954. Every effort has been made for this high voltage electron microscope to be not only versatile in applications but also convenience in operation and maintenance. Thus a prototype of the 300 kV microscope was set up in 1957. Its inconveniences in operation were eliminated during the test run in 1957-1961. And in 1962, it was reconstructed in the form of the present apparatus.*

Several quantitative measurements about the effects of high voltage electrons to the specimens were carried out for the first time by this electron microscope. The instrument has been used almost daily in the studies of various materials by the associated members and many visiting researchers.

2. 300KV HIGH VOLTAGE GENERATOR

The high voltage generator consists of three 60 cps transformers set up in cascade and can produce any voltage in the range of 75-300 kV. The generator has a stabilized input but has no feed-back system. The complete circuit is shown in Fig. 1. A part of the generator is shown in Fig. 2. As can be seen in these figures, the generator

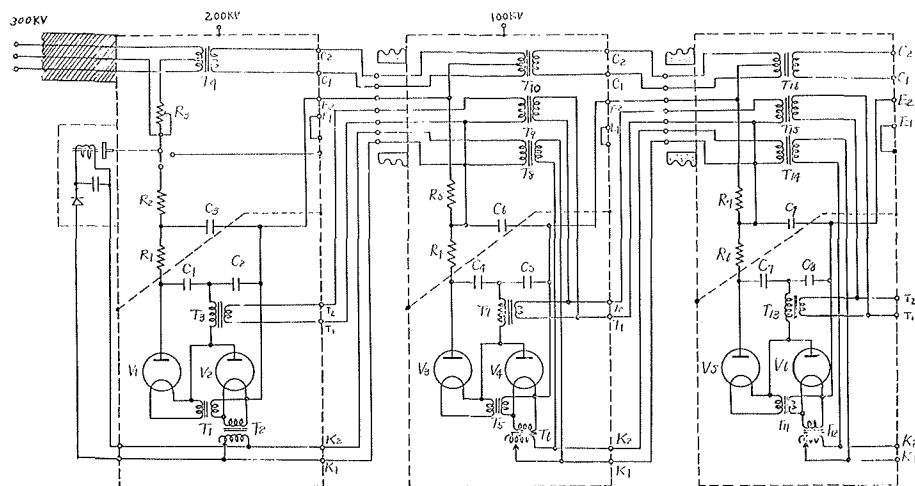


Fig. 1. Diagram of high voltage generator of 300 kV electron microscope.

* The outline of the instrument before this reconstruction is found in the previous reports of the present authors.

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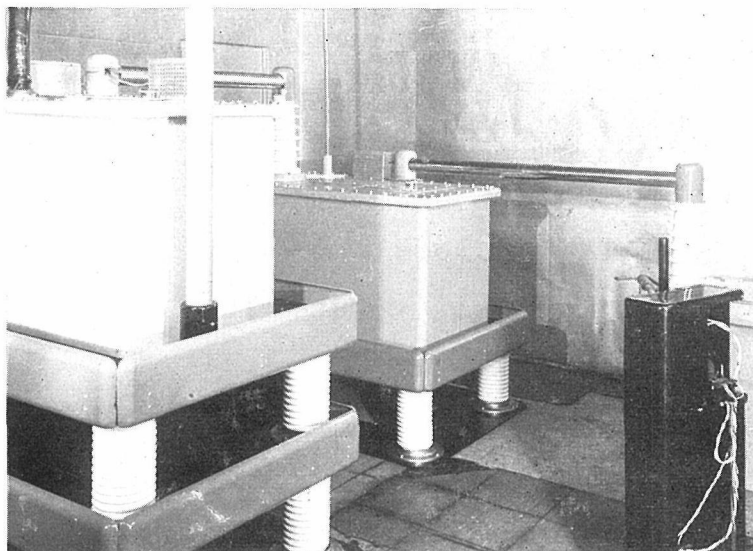


Fig. 2. Photograph of the high voltage generator. The three tanks are for 100, 200 and 300 kV stages, respectively. The outside wall of these oil tanks are maintained at 0, 100 and 200 kV in the same order as above. The black cable on the left-top of the figure contains the lead wire for 300 kV. The black tank on the right is a remote-controlled discharger.

consists of three insulated 100 kV d.c. sets, that is, each unit provides an insulated primary supply voltage for the next unit. Filament power is supplied through three filament transformers connected in series, each of which is insulated for 100 kV. The necessary stability of output voltage is ensured by using large smoothing capacities and resistors (0.05 micro farad and 5 megohms). The output stability expressed in terms of the ripple of the output voltage of each stage is 8.4×10^{-5} (1st stage), 6.3×10^{-5} (2nd stage) and 10×10^{-5} (3rd stage). The voltage of input is stabilized within $3 \times 10^{-4}/\text{min.}$ by a magnetic and an electronic stabilizers.

The potential of 300 kV is connected to the electron gun through an insulating cable without exposing it to the open air. Through the metal net covering the cable surface, the 200 kV potential is supplied to the second set of electrodes in the accelerator. The diameter of the insulating cable is 65 mm which is large enough to eliminate corona discharge at 200 kV. The 100 kV potential is fed through an aluminium pipe with an appropriate diameter. The voltage of primary input is remotecontrolled by a motor-driven variable auto-transformer. The final composite stability at about 300 kV is estimated to be within $3 \times 10^{-4}/\text{min.}$ from the fact that a fine lattice spacing of 12.5Å can be resolved.

The residual high potential stored in the capacities is discharged by the contact of motor-driven high resistor rods as shown in Fig. 2.

3. THE ELECTRON GUN AND ACCELERATOR

A photograph of the microscope is shown in Fig. 3 and its cross-sectional scheme

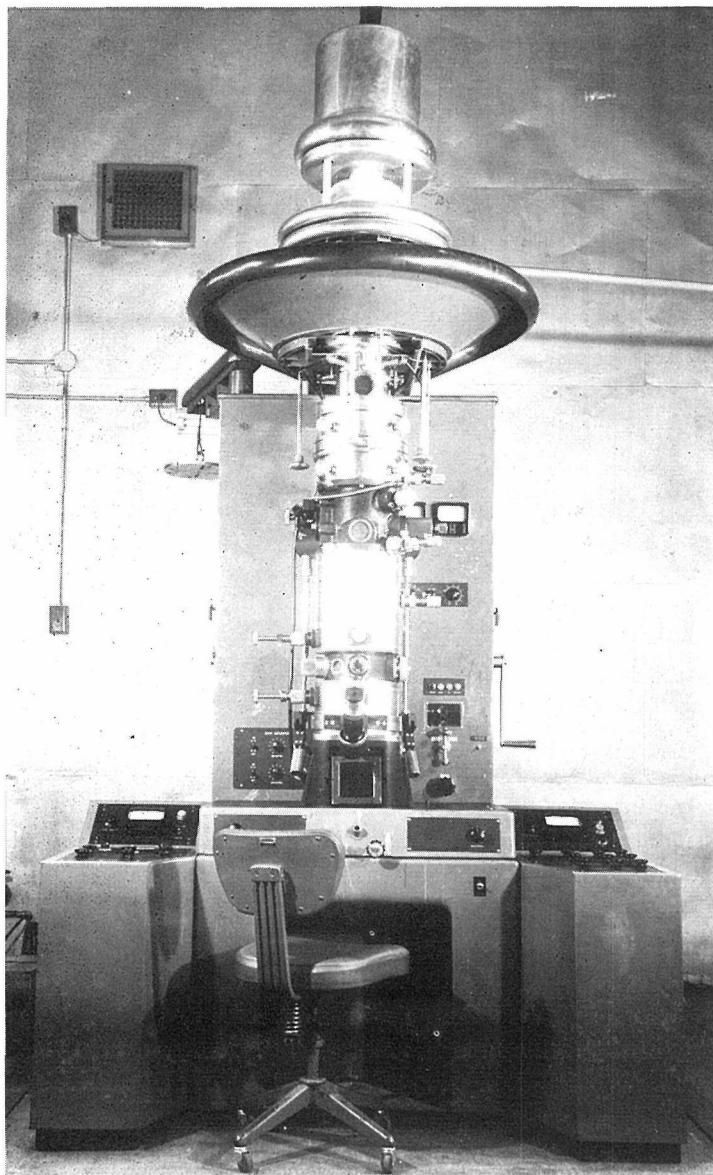


Fig. 3. Photograph of 300 kV electron microscope.
The bell-shaped cylinder on the top is the corona cap and the lower conical disk is the x-ray shield. The derrick which appears on top-left of the frame is used for demounting the microscope column.

is shown in Fig. 4. The electron gun consisting of three electrodes is mounted above a large dish-shape x-ray shield. The first top electrode at 300 kV (the electron source) is connected to the cable head which is insulated in the vacuum from the second (200 kV), while the latter and the third electrodes (100 kV) are installed in two insulating porcelain cylinders of 30 cm height each and connected with the corresponding leads

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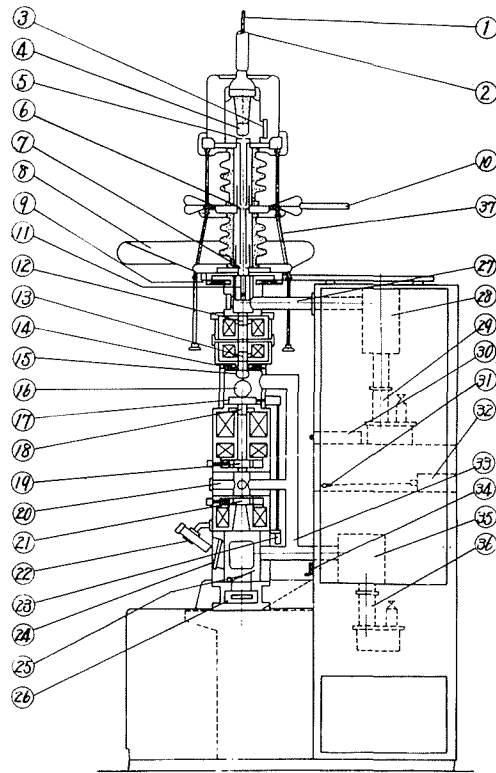


Fig. 4. Cross sectional diagram of 300 kV electron microscope.
 ①: Feeder line for 300 kV, ②: Feeder line for 200 kV, ③: Window for filament replacement, ④: Cathode, ⑤: The first anode, ⑥: The second anode, ⑦: The third anode, ⑧: x-ray shield, ⑨: The first beam deflector, ⑩: Feeder line for 100kV, ⑪: x-ray shield, ⑫: The first condenser lens, ⑬: The second condenser lens, ⑭: The second beam deflector, ⑮: Isolation-vacuum flap valve, ⑯: Air lock for specimen exchange, ⑰: Specimen chamber, ⑱: Objective lens and aperture, ⑲: Intermediate lens, ⑳: Specimen chamber for electron diffraction of high resolution type, ㉑: Projector lens, ㉒: Optical magnifier, ㉓: Specimen migrator, ㉔: Viewing port, ㉕: Fluorescent screen, ㉖: Photographi cplate, ㉗: Evacuating pipe line for gun, ㉘: Cold trap, ㉙: Oil diffusion pump, ㉚: vacuum gauges (ionization and thermocouple type) ㉛: Discharge tube, ㉜: Transformer for discharge tube, ㉝: Evacuating pipe line for microscope column, ㉞: Controller for vacuum valves, ㉟: Vacuum valves, ㊱: Oil diffusion pump, ㊲: Insulated handle for the alignment of gun.

exposed to the air. These electrodes form an electrostatic lens system. By the preliminary experimental measurements carried in an electrolytic tank, the dimensions of the electrode system were determined; the focal lengths of the first, second and the third electrodes converging electron beam have been estimated to be 12mm, 245mm, and 720mm respectively. The image of the electron source by this electrostatic lens

system is formed on a plane just below the specimen position. The gun system can easily be removed from the main column by means of two guide arms attached to the housing case of the evacuating system behind the column. The filament can be replaced very quickly by taking out the filament assembly block through a window on the side wall of the gun.

Each stage of the accelerator can be aligned on the optical axis by the fine shift of each electrode. For this adjustment two sets of cams driven by insulating polymer rods are provided at the top and second stages of the accelerator. Since the cascade system of high voltage generator is open, it is very easy to apply high tensions to a single section or two sections of the accelerator. This leads a very convenient operational technique for the re-alignment after cleaning or repairing.

4. THE MICROSCOPE COLUMN

Since the very fine tilting or traverse of the tall and heavy accelerator system as a whole is very difficult, two sets of magnetic beam-deflectors are inserted between the gun and the specimen chamber. They are located at the top and the bottom of the condenser lenses. Thanks to these deflectors following operational conveniences are available. First, the compensation of the deflection of the electron beam due to the geomagnetism is very easy, that is, there is no necessity of the re-adjustment of the accelerator electrodes even when the accelerating voltage is changed to a large extent i.e., 100~300 kV. Secondly, the centering of narrow incident beam to the specimen can be done very precisely. And lastly the oblique illumination can be applied without any mechanical tilt of the electron gun, which is otherwise impossible for such a large accelerator.

Using the double condenser system, the minimum diameter of the illuminating electron beam can be reduced as small as 1 micron when projected on the specimen.

The objective lens is followed by intermediate and final projector lens. The diameter of the iron shield for each lens is 250mm. The ampere-turns of each lens are tabulated in Table 1.

Table 1.

1st condenser lens	0~3800 amp·turns
2nd condenser lens	0~2000 amp·turns
Objective lens	0~5600 amp·turns
Intermediate lens	0~3300 amp·turns
Projector lens	0~4000 amp·turns

The maximum total magnification is $100,000\times$ at 100 kV and $40,000\times$ at 300 kV. The maximum magnification is limited by the maximum lens power of the projector for the first order imaging. The objective lens is water-cooled and has an adjustable diaphragm and a magnetic stigmator. The water-cooling is effective not only to prevent the temperature rise of the lens coil but to keep the temperature of the specimen stage constant during some prolonged observations. And the pole-piece assembly of the objective is interchangeable with a long focus one by replacing it through two

large windows on both sides of the specimen chamber. Since the optical axis of the objective is fixed, the alignment of the imaging system on this axis is performed by moving the pole-pieces of the intermediate and projector lenses. These pole-pieces and all the apertures are interchangeable and can be pulled out from the path of electron beam without breaking the vacuum.

The specimen chamber has a large room and the heating or cooling specimen stage is available. In the case of such a special stage, a long focus lens has to be used for the observation. The specimen can be exchanged through the air lock mechanism when either ordinary stage or tilting device (up to $\pm 20^\circ$) are used.

Another specimen chamber with air lock is placed between the intermediate and the projector lenses in order to observe the high resolution electron diffraction pattern and shadow microimage¹⁰⁾¹¹⁾ with a goniometer specimen holder. The index of resolution for this procedure is estimated to be $2 \cdot 10^{-5}$.

The camera lengths for various diffraction techniques are as follows: a) 85cm when the specimen is placed upon the objective lens with a hole wide enough to pass the diffracted beams and the pole-pieces of the intermediate and projector lenses are displaced out of the path. In this working condition it is easy to realize the high resolution diffraction patterns by means of the double condensers, b) 35cm for the selected area diffraction at 300 kV, and this distance can be made doubled by the use of long focus objective, c) 50cm for the ordinary work at the lower specimen chamber, and d) convertible as a very long focal camera when the variable magnifying lens system is applied.

To make easy the setting up or dismantling of each heavy sectional block of the column, a special derrick is installed on the top of stand frame. And the hoist is driven by a large handle as shown on the right side of the housing of evacuating system in Fig. 3 and the arm can be swung aside.

In order to eliminate x-ray radiation from the instrument, all observation windows are made of lead glass whose thickness is equivalent to 4mm of lead. The stray x-ray outside the microscope was measured with an x-ray survey meter to be less than 5mr/hr., that means, less than the maximum dose rate permitted by Japanese regulations.

This nearly perfect prevention of x-rays from the microscope was attained by a special consideration given for the elimination of the targets which acted as the strong emitters of x-rays. In the prototype of this microscope an aperture acting as anode was inserted just below the accelerator. This aperture was the strongest x-ray source even when the hole was wide enough to pass the central cone of the electron beam. And the aperture was apt to melt when deflected beams were stopped by it.

As the lens design of accelerator was improved into the present form, the divergence of electron beam was reduced and the necessity of an anode aperture, limiting the beam incident to condenser lens was diminished. Thus the present microscope has no anode aperture, and the narrow direct beam is diverged by the strong lens of the first condenser into very wide angle and the x-rays generated from the inner surface of the condensers which is covered with copper cylinder, are stopped perfectly by the thick windings of lens coils and their thick iron shield. The x-rays emitted from the aperture of the second condenser are very weak because the incident intensity of

electron beam to this aperture is largely reduced by the first condenser.

In the first step of alignment of the electron gun by inspecting the direct beam spot on fluorescent screen, the applied high voltage is limited lower than 200 kV, and the objective lens is used as the scatterer of direct electron beam. It is to be remembered that the optical axis of the objective is chosen as the reference of the whole optical system.

For the sake of these precautions, this microscope is guaranteed to be safe for the operators to work without any x-ray protectors.

5. VIEWING CHAMBER AND CAMERA

The viewing chamber has three wide windows made of lead glass. The fluorescent screen is 15cm \times 10cm. For the closer inspection of the image projected on the screen, a telescopic magnifier with a large aperture objective is provided through the ball of a ball-socket mount. And the supporting arm of the telescope can be rotated around the column. At the magnification of 3 times, the whole area to be photographed is covered within the field of view of this telescope. A large magnification ($10\times$) is attained with the interchangeable eyepiece, and in this case, the diameter of the effective exit pupil is 4 mm which assures the magnified image being as bright as the direct observation.* A beam stopper and a Faraday cage which can be set at any point in the plane perpendicular to the electron beam are inserted to the viewing chamber. The Faraday cage is used for intensity measurements of the direct electron beam as well as that which has passed through the specimen. A sensitive d. c. ampere meter (10^{-13} A) is required for the measurement of the intensity of image forming electrons at a high magnification. The camera holds two photographic plates of 6cm \times 16.5cm mounted end to end in a long holder, giving 6 exposures of 5 cm \times 5 cm size or 12 exposures of half size.

The continuous record of the change of diffraction patterns by heating, cooling, or electron bombardment can be made easily by moving the long photo plate.¹²⁾

6. EVACUATION SYSTEM

The column is divided into two parts by a flap-valve inserted just below the condenser system, i. e., the electron gun is separated by this valve from the lower part of the column. Each half has its own pumping system. By isolating the vacuum with the flap-valve, the high tension being applied to the gun can be kept on throughout the operation except for the accidental filament exchange. This leads to a high operational efficiency and gives a reproducibility of the applied accelerating voltage.

Cold traps are provided above the diffusion pumps; however, there is no necessity of using them for ordinary works, because the contamination deposited on the specimen is so small compared with conventional electron microscope. Even the contamination exists, it is much less impeding for the transmission of high voltage electrons.¹³⁾

The third mechanical pump is provided for evacuating the air locks on the two

* This type of telescopic magnifier was originally developed in 1951 by one of the present authors (K.K.) to be installed onto the conventional electron microscopes made by Shimadzu Seisakusho Ltd.

specimen chambers and prepumping of photographic plates in a separate vacuum chamber. A foot-switch is used to turn off the backing pumps during the exposures for photographing.

7. ROOM FOR INSTALLATION AND ITS AIR-CONDITIONING

The room which houses the microscope is 5.5m high and air conditioned to keep the temperature at 20~25°C and the relative humidity at around 60%. Although the room needs to be specially equipped, the present system with semi-open high voltage parts is very simple and easy to be reformed for any experimental trial. For the sake of high voltage supply through a coaxial cable, there appears no detectable corona even when the room is not sufficiently air-conditioned.

The air-conditioning is necessary only for the prevention of accidental leakage or breakdown through the surface of the porcelain insulators affected by the relatively high humidity in Japan. Each porcelain insulator is loaded with only 100 kV and the maintenance of very low room humidity is not required.

Another 300 kV electron microscope developed parallel to this one by the Hitachi group is equipped with an open van de Graaf static generator as its high voltage supplier, and must be operated in a very dry room conditioned at as low as 59% or less in relative humidity.

An old 300 kV electron microscope, which was trially built by RCA group, was suffered from corona discharges grown from minute dust (presumably fibrous) particles absorbed on the connecting pipe for 300 kV. As the high potential part of this microscope exposes only 200 kV to the air, no detectable corona discharge appears, and its high voltage generator with large time constant is very stable for the minute discharges possibly to occur. Hence no precaution is taken to eliminate fibrous dust from the air.

All the inner surfaces of the installation room are covered with metal plates and grounded to prevent the possible charging up of any electrically floating subjects. The main part of the microscope is set on a heavy block of vibration damper which is balanced to the lowered center of gravity of the whole system and is suspended by elastic rubber cylinders at its upper corners. No special lifting device is provided on the ceiling of the room, since, as mentioned already, the accelerator is removable aside by the sliding guide and a swing-out derrick on the stand frame for the dismantling or re-constructing of the main column is equipped.

8. STABILITY AND RESOLVING POWER OF THIS MICROSCOPE

The prototype of this instrument showed a poorer resolution for thin specimen compared with that of the conventional electron microscope. Drift of high voltage power supply was estimated to be less than as 3×10^{-4} /min. And the alternating current component (ripple) of the output voltage, which arises dominantly from the induction from 60 cps power line in this case, was measured to be in a magnitude of larger than 2×10^{-4} . In order to reduce the induction from a. c. parts and to protect the step-up transformers from leakage and breakdown along the surface of their

secondary coil, the unit high voltage step-up generators were reconstructed in the present form as shown in Fig. 2. The rectifying circuit was replaced with a double voltage rectifying system which made the output voltage of each main transformer one-half

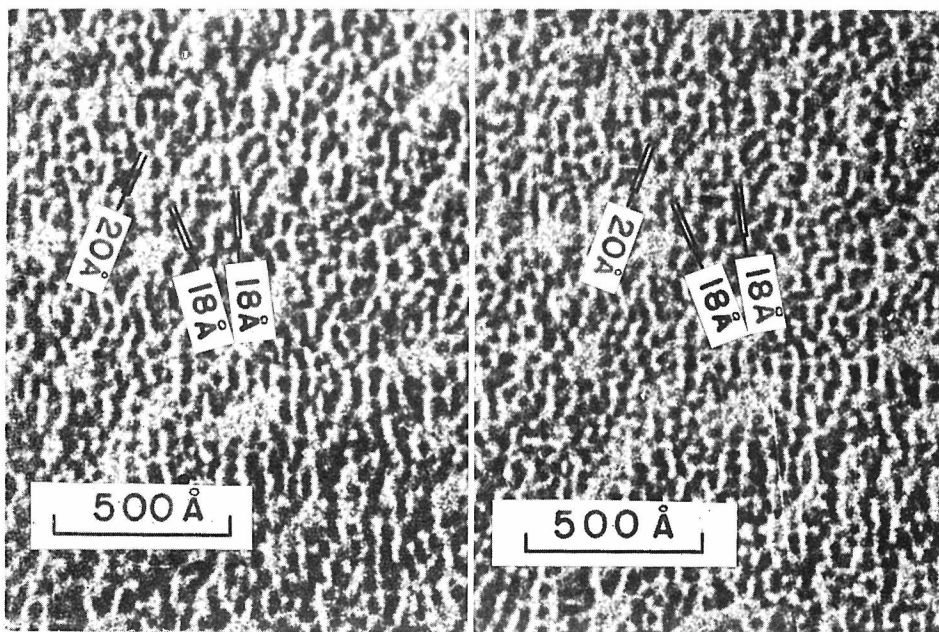


Fig. 5. Reproduction test of resolution at 300 kV. Specimen: vacuum evaporated particles of Pt-Pd



Fig. 6. Lattice image of copper-phthalocyanine crystal taken at 300 kV, which shows 12.5 Å spacing.

of the original system, and the more effective electrostatic shields could be provided between a.c. and d.c. circuits, because the dimensions of every parts were reduced so much that there remained enough space to install the shields.

As the result of this improvement, the a. c. component of the out-put was reduced to one-quarter of the original model, and thus the resolving power of this microscope was greatly enhanced up to a grade comparable with the conventional electron microscopes.

Fig. 5 is a set of successive records of the images of vacuum evaporated Pt-Pd particles made with the original high voltage power supply. The highest resolving power of the prototype was estimated to be around 18\AA at 300 kV.

Fig. 6 shows a lattice image of Cu-phthalocyanine crystal observed under the microscope with the improved power supply, and from this the resolving power for lattice image is proved to be not poorer than 12.5\AA at 300 kV. This result also proves that the final stability of the cascade high voltage generator has to be less than $3 \times 10^{-4}/\text{min.}$, when the chromatic aberration due to the fluctuation of accelerating voltage is considered to be the dominant factor limiting the resolution.

9. EFFECTS OF HIGH VOLTAGE ELECTRONS REVEALED BY THIS MICROSCOPE

Several quantitative studies of the effects of high voltage electrons upon various kinds of specimen were carried out for the first time with this 300 kV microscope. The results were reported in other papers. Two examples are illustrated here to show the effects on transmissive power and in radiation damage of the specimen.

Fig. 7 shows a comparison of the change in the numbers of thickness fringes observed at the same area of a wedge-shaped aluminium foil with 100 kV, 200 kV and 300 kV electrons. The numbers of fringes detectable on the original plate are 7 for 100 kV, 13 for 200 kV and 17 for 300 kV. Since the equivalent thickness of specimen corresponding to a single fringe increases with the accelerating voltage, it is clear that the increment of maximum thickness for transparency is estimated to be larger than that of the number of fringes mentioned above.

For this relation of the transmissive power of electrons, the effect of relativity must be taken into consideration. However, in the range up to 300 kV, a definite conclusion is hardly available because the effect of the growth in mass, which is to overcome the effect of the reduction of wave length is much slighter than that in the region over 500 kV.

As to the damage of specimens induced by the electron bombardment, an example of conspicuous effects of increased accelerating voltage is shown in respect to a polymer crystal. Fig. 8 is a unique image of a spirally grown single crystal of polyoxymethylene observed with 250 kV electrons. In this photomicrograph triple effects of high velocity electrons upon polymer substances are realized simultaneously. First, the sharp moiré patterns observed all over the illustrated area indicate that the molecular arrangements in every layer are kept intact during the electron bombardment. Secondly, the transmissive power of the applied electrons is so high that the superposed multiple layers, more than 30 at the thickest part, appear transparent. And thirdly, the resolution

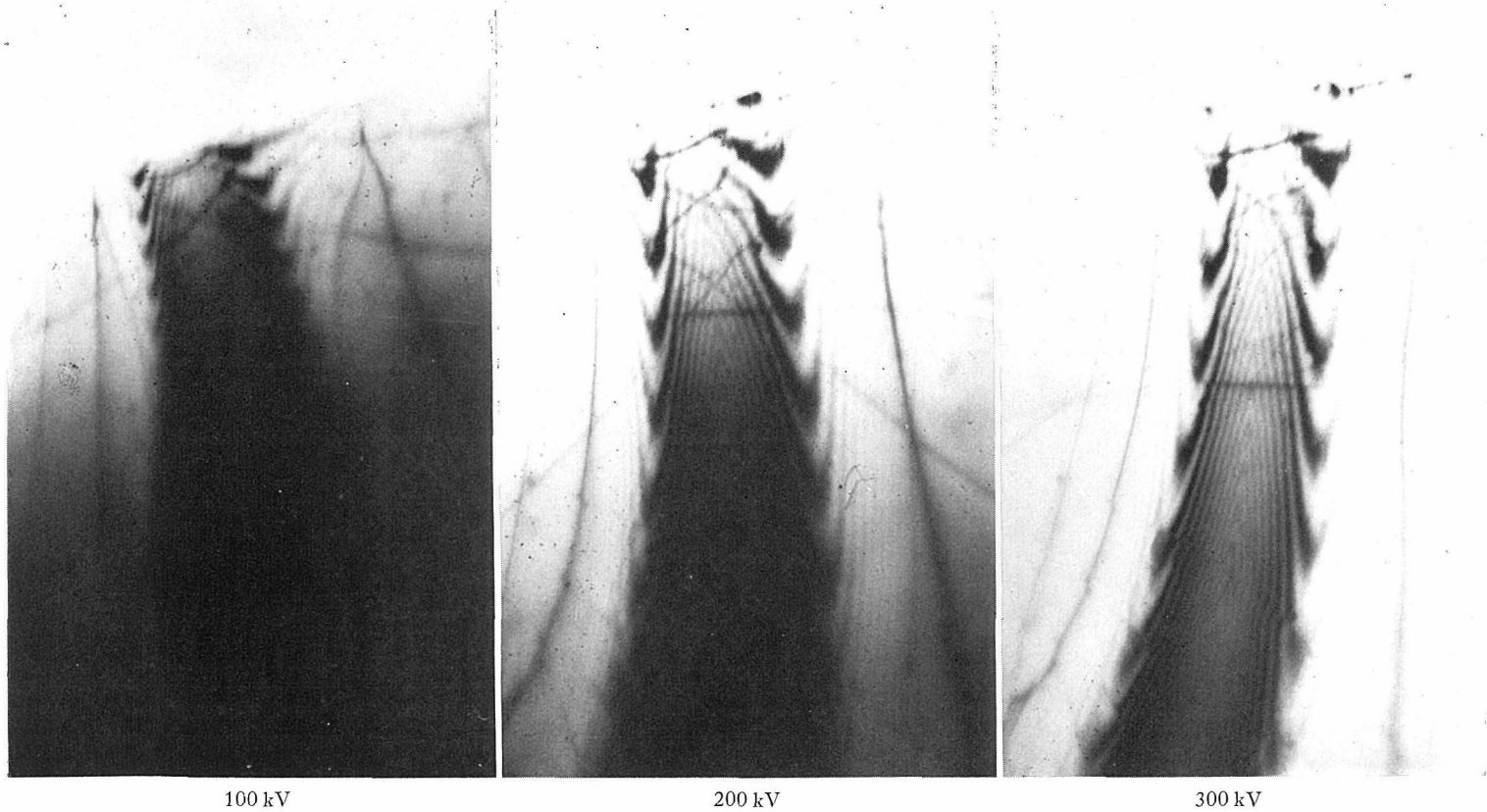


Fig. 7. Comparison of transmission of electrons at various accelerating voltages.
Specimen: electro-polished Al-wedge.
Thickness fringes are due to the (111) reflection.

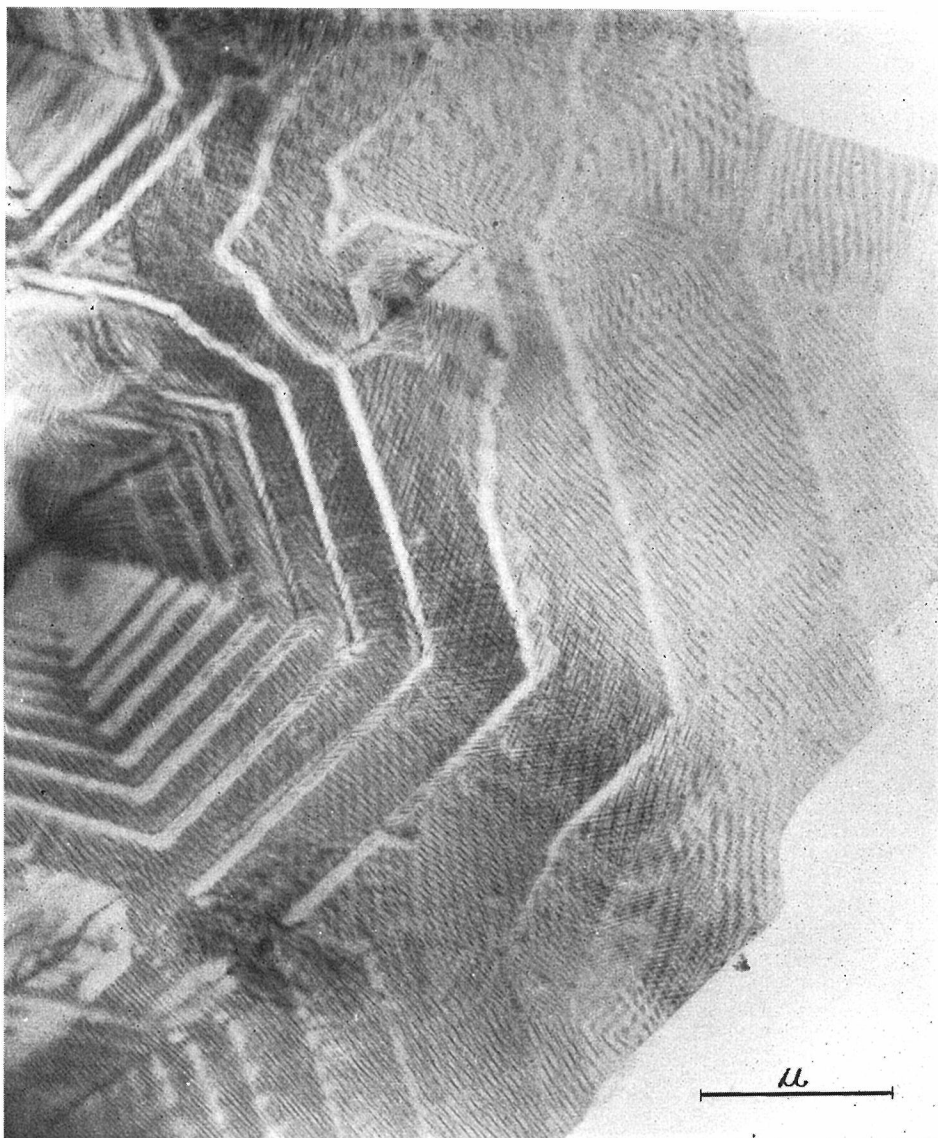


Fig. 8. Complex moiré patterns observed in a spirally grown laminar crystal of polyoxymethylene. The chain molecules are arranged perpendicular to the laminar face of the crystal.

at the thickest region of the specimen is not inferior so much compared with the very thin part (each lamella has an equal thickness of around 100\AA). The complexity of moiré patterns can be ascribed to the effect similar to that of multiple beam interference. It is detected by close examination of this patterns that weak lines corresponding to submaxima between sharp strong lines are appearing where more than 2 layers are superimposed. It has to be emphasized that electrons accelerated at a lower voltage (upto 100 kV) have never produced such an image with good definition and high contrast as illustrated here.

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* After the completion of the manuscript of this paper, the present authors had the opportunity to read a copy of the proceedings of AMU-ANL High Voltage Electron Microscope Meeting, Argonne (1964). In this proceedings, E. Zeitler and G. F. Bahr who were the visiting researchers to this Institute, contributed a paper dealing with their results obtained with this 300kV electron microscope.

A brief reference to the reduction rate of contaminations on the specimen was made by them in their paper. According to their results, the contamination was so small as one fifth when the accelerating voltage was varied from 100kV to 200kV.